Characterization of Machine Variability and Progressive Heat Treatment in Selective Laser Melting of Inconel 718

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Abstract

The absence of an economy of scale in spaceflight hardware makes additive manufacturing an immensely attractive option for propulsion components. As additive manufacturing techniques are increasingly adopted by government and industry to produce propulsion hardware in human-rated systems, significant development efforts are needed to establish these methods as reliable alternatives to conventional subtractive manufacturing. One of the critical challenges facing powder bed fusion techniques in this application is variability between machines used to perform builds. Even with implementation of robust process controls, it is possible for two machines operating at identical parameters with equivalent base materials to produce specimens with slightly different material properties. The machine variability study presented here evaluates 60 specimens of identical geometry built using the same parameters. 30 samples were produced on machine 1 (M1) and the other 30 samples were built on machine 2 (M2). Each of the 30-sample sets were further subdivided into three subsets (with 10 specimens in each subset) to assess the effect of progressive heat treatment on machine variability. The three categories for post-processing were: stress relief, stress relief followed by hot isostatic press (HIP), and stress relief followed by HIP followed by heat treatment per AMS 5664. Each specimen (a round, smooth tensile) was mechanically tested per ASTM E8. Two formal statistical techniques, hypothesis testing for equivalency of means and one-way analysis of variance (ANOVA), were applied to characterize the impact of machine variability and heat treatment on six material properties: tensile stress, yield stress, modulus of elasticity, fracture elongation, and reduction of area. This work represents the type of development effort that is critical as NASA, academia, and the industrial base work collaboratively to establish a path to certification for additively manufactured parts. For future flight programs, NASA and its commercial partners will procure parts from vendors who will use a diverse range of machines to produce parts and, as such, it is essential that the AM community develop a sound understanding of the degree to which machine variability impacts material properties.

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Introduction

Additive manufacturing (AM) encompasses a broad swath of technologies that consist of "joining materials to make objects from three-dimensional model data, usually layer by layer, as opposed to subtractive manufacturing methodologies" [1]. AM first emerged in the 1980s with stereolithography and has since grown to include fused deposition modeling (FDM), selective laser melting (SLM), selective laser sintering (SLS), electron beam melting (EBM), laminated object modeling (LOM), and laser engineered net shaping (LENS) [2]. While there is no formal recognized classification schema for AM, processes can be broadly categorized according to 1) the technique used to deposit the layers, 2) the energy source/method by which the deposited layers are fused together, 3) the state of the starting materials (liquid, wire, filament, powder, etc.) and/or 4) the type of material (biomaterial, polymer, metal, composite, ceramic) [1]. The level of activity in AM research has burgeoned in recent years as the field has become a national priority in science and technology policy. The potential of AM to benefit multiple sectors, return manufacturing expertise to US soil, and promote future industry has been recognized by the Obama administration, the Office of Management and Budget, and the Office of Science and Technology Policy [3]. Currently, government directed initiatives such as America Makes and the Materials Genome Initiative (MGI) work to facilitate collaboration in materials research and transfer knowledge among industry, academia, and government agencies as well as develop data management tools (such as informatics databases) that will accelerate the development (and subsequent adoption by industry) of new materials and manufacturing techniques [4]. AM represents a large focus of these efforts.

NASA Marshall Space Flight Center in Huntsville, Alabama has been at the forefront of additive manufacturing research since 1991, when the additive manufacturing laboratory was established with two primary purposes: 1) to provide a rapid prototyping capability for spaceflight hardware and 2) develop methods to build parts in-space using material extrusion techniques such as FDM. In subsequent years, the laboratory became part of a government/industry consortium – the National Center for Manufacturing Science (NCMS) -- with an emphasis on the development of metallic additive manufacturing systems. In recent years, the laboratory's has focused on maturing metallic AM techniques to produce rocket propulsion hardware, specifically the qualification of electron beam and powder bed fusion processes for this application.

As two decades of innovation in materials and processes have transitioned AM from a laboratory curiosity to a manufacturing technique capable of fabricating production quality parts, the aerospace and aviation sector has begun to adopt AM and integrate it into their design processes. The benefits of AM for propulsion components, which generally have long lead times

and large costs when produced by traditional subtractive methods, is widely recognized. For instance, an additively manufactured gas generator duct for NASA's J-2X engine imparted a 70% reduction in cost and a 50% reduction in schedule/manufacturing time over its conventionally manufactured counterpart [5]. For the flex joint on the RS-25 engine, the selective laser melting (SLM) process was able to reduce the part count from 45 (heritage design) to 17 and the number of welds from 70 to 26 [5]. These examples illustrate that AM is a particularly attractive option for the low-rate production, complex hardware geometries that are characteristic of propulsion hardware.

While industry and NASA are eager to capitalize on AM's ability to reduce part counts, minimize the number of welds and brazes, and impart schedule and costs savings, no new technology is adopted without challenges. There are four key technical hurdles that NASA and the broader aerospace sector must address as we work together to develop and certify AM parts and processes for use in spaceflight (and particularly human-rated spaceflight) systems [6]:

1) Materials characterization – understanding the influence of AM build parameters, powder characteristics, thermal processing, and surface texture on the material properties of the end-use part
2) Standard design practices – development of guidelines for mechanical design, testing specifications, management of uncertainties associated with material properties and operational environments
3) Process modeling, monitoring, and control – physics based modeling of transport phenomena in AM processes, in situ defect detection, prediction of material properties based on material characteristics and build parameters
4) Establishing flight certification logic – development of a part classification system using a risk-based approach, understanding of part failure modes and establishment of NDE and fracture control requirements, approaches to lot acceptance testing

The additive manufacturing laboratory at NASA Marshall and its partners have significant ongoing activities in all of these areas. The study presented in this paper focuses on characterizing the effect of machine variability on additively manufactured materials. This work is most closely tied to area (1) (materials characterization and understanding of process/property relationships), but is also key to standard design practices (since designers and analysts need to know the degree of variability in material properties, specifically the minimum characteristic material property values used for design, that can be expected when identical parts are produced on different AM machines), process modeling and control (as a smart developer and procurer of AM parts, we need to understand what factors contribute to machine variability effects and the procedures/controls that most effectively minimize variation in properties among end-use parts),

and flight certification (property variability, whether a consequence of machine differences or other process factors, influences the selection and development of procedures for lot acceptance testing).

Process Variability in AM

The design flexibility and cost/schedule savings enabled by AM make this class of technologies particularly attractive for production of aerospace components. However, the use of AM processes for production of aerospace grade materials rather than prototyping is still regarded as nascent. One of the central challenges of AM implementation in industry is to identify, quantify, and control the variables that can influence the material properties of the end-use part. Designers and analysts rely on statistically based minimum material properties to establish safe margins and ensure that a part will be able operate in its intended use environment. These properties are usually characterized through a material allowables program and subsequently published in materials handbooks such as MMPDS (Metallic Materials Properties Development and Standardization) and CMH-17 (Composite Materials Handbook 17). As an emerging technology, AM materials and process (M&P) specifications do not exist to the same level of fidelity and control as those established in MMPDS, CMH-17, and similar governing M&P documents recognized by government and industry for conventionally manufactured materials [7]. For aerospace, in many cases AM parts are regarded as process specific and certification activities are undertaken on a part by part basis.

In lieu of standards, organizations who use AM in production and/or critical hardware may undertake their own small-scale allowable programs to explore processing conditions and characterize the effect of process variables on material properties with the ultimate goal of using this knowledge to establish minimum property design values that serve as critical inputs to designers and analysts. In addition to a reduced sample size compared to the recommendations of MMPDS and CMH-17, another limitation of these programs is that (in most cases) specimens or parts are produced on a single AM machine. Previous experience has demonstrated that variation in material properties may not only be sensitive to process variables, but specific to the machine the specimens were produced on. There is a concern from a process control standpoint that design minimum properties might be slightly different when AM parts are produced on different machines, even if the build parameters, materials, thermal processing conditions, etc. remain the same.

There is little in the published literature assessing the machine specificity of AM parts. While there is some speculation that powder bed fusion processes may be more susceptible to machine variability effects than AM processes that are extrusion based, there are no formal studies to substantiate this. Based on the MSFC laboratory's experience with powder bed fusion techniques, it is possible for two machines operating at identical parameters with equivalent base materials to produce specimens with slightly different material properties. Quantifying the impact of machine variability on material properties and developing process controls to minimize these effects are key to establishing AM methods as reliable alternatives to traditional subtractive manufacturing [8]. Understandably, there is some resistance among the standards community toward the inclusion of emerging technologies such as AM in design handbooks due to process variability -- manufacturing processes must demonstrate that they are highly repeatable, reliable and consistent before their incorporation into standards documents can be considered. An understanding of machine variability in AM will accelerate the technology's commercial acceptance and create a knowledge base that is critical to transitioning AM from the custom LRP (low rate production) sphere it currently occupies to the world of mass HRP (high rate production), where production parts are fabricated at a rapid rate with confidence that they will meet or exceed all stringent functional requirements for spaceflight hardware.

Characterization of machine variability is critical to NASA, academia, and the industrial base as we collaborate to establish a path to certification for additively manufactured hardware. For future flight programs, NASA and its commercial partners will procure AM components from vendors who use a diverse range of machines and AM equipment to produce parts. A sound understanding of the degree to which machine variability impacts material properties is essential for lot acceptance and hardware certification.

Methodology

In an effort to address the knowledge gap related to the effect of machine variability on material properties, a small-scale study on this subject was undertaken at NASA Marshall Space Flight Center. The advanced manufacturing team at NASA MSFC operates two Concept Laser Selective Laser Melting (SLM) machines. Although the material group for this investigation considered only the nickel-based alloy Inconel 718, these machines are also capable of processing high grade steels, Aluminum alloys, Titanium alloys, and pure Titanium. The M1 machine is best-suited for the production of small to medium sized components. The build envelope for the M1 is 250 x 250 x 250 mm (x,y,z) and the laser system is a fibre laser capable of providing up to 400 W of energy. The M2 from Concept Laser is also a 400 W laser system but has a slightly larger build box (250 x 250 x 280 mm). The M1 and M2 were used to produce

60 tensile specimens (30 from each machine) at identical default machine parameters using virgin Inconel 718 powder from the same lot.

The study had two major objectives:

1) To determine the amount of variability in mechanical material properties between the two machines
2) To determine the effect of heat treatment on machine variability

The 60 Inconel 718 round tensile specimens were tensile tested in accordance with ASTM E8, "Standard Test Methods for Tension Testing of Metallic Materials" [9]. The specimens were built in the vertical (Z) orientation. The machined specimens had a nominal diameter of 0.250 inches and a nominal length of 3.0 inches. The environment for the testing was lab air at room temperature, approximately 70 degrees Fahrenheit. Tests were conducted in displacement control at an initial rate of 0.02 in/min. This rate was maintained up to 2% strain, after which the rate was increased to the final displacement rate of 0.05 in/min. Stress data were obtained from load measurements from a calibrated 20,000 pound load cell and pre-test specimen dimension measurements. Strain data were obtained from displacement measurements from a 1.00 inch extensometer calibrated to 50% strain. Ultimate stress, yield stress, and fracture elongation values were determined from the resulting stress-strain curve according to procedures outlined in ASTM 38. Elastic modulus was reported for reference, but specific modulus tests are needed for true values. The following material properties were reported for each of the sixty specimens: Ultimate tensile strength σ_{UTS}, yield strength σ_Y, elastic modulus E, fracture elongation e (expressed as a percent), and reduction in area A_R (also a percent).

All specimens were stress relieved at 1950 F +/- 25 F for 1.5 hours, -5/+15 min., followed by a 2-4 Bar argon quench. The stress relief process is necessary to separate the samples from the build plate. Forty specimens (20 from each machine) underwent a hot isostatic press (HIP) treatment at 2050 +/- 25 F and 15,000 +/- 250 psi for 3 hrs. +/- 15 min. Twenty of the HIP specimens (10 from each machine) were heat treated according to AMS 5664E, which consists of a solution heat treatment at 1900-1950 F +/- 25 for a time commensurate with cross-sectional thickness, and cooling at a rate equivalent to an air cool or faster, followed by a precipitation heat treatment at 1400 F +/- 15 F until a total precipitation hat treatment time of 20 hrs. has been reached, then cooled.

To facilitate comparative analysis, the data were grouped according to machine and postprocessing conditions. The group name, number of specimens in the group, the SLM

machine (or machines) used to build the specimens, and the post-build conditioning that correspond to the data groups are summarized in Table 1.* The machine used to produce the specimens and the post-build conditioning for a group can be identified from the group name. For instance, M1 SR+HIP refers to the group of specimens that were produced on the Concept Laser M1 and underwent stress relief followed by hot isostatic press.

Table 1 Summary of specimen groups

Specimen group name	Number of specimens	SLM machine(s)	Post-build conditioning
M1 SR	10	M1	SR
M2 SR	10	M2	SR
M1 SR+HIP	10	M1	SR+HIP
M2 SR+HIP	10	M2	SR+HIP
M1 SR+HIP+HT	10	M1	SR+HIP+HT
M2 SR+HIP+HT	10	M2	SR+HIP+HT

While Table 1 represents the groupings that are the most useful for direct comparative analysis of material properties, we can also construct pooled data groups. These groupings are comprised of one of the following: a) samples which were subject to the same postprocessing conditions but were built on different machines or b) specimens built on the same machine but which underwent different postprocessing regimes. The entire set of 60 samples are analyzed in the M1+M2 data group.   The pooled data groupings appear in Table 2.

*Acronyms are used to designate post-build conditioning. SR indicates the specimens underwent stress relief, HIP corresponds to hot isostatic press, and HT is heat treatment (AMS 5664E). For example, a specimen group identified by SR+HIP+HT indicates that the specimens were stress relieved, underwent HIP, and then heat treatment per AMS 5664E.

Table 2 Summary of pooled specimen groups.

Specimen group name	Number of specimens	SLM machine(s)	Post-build conditioning
M1+M2	60	M1, M2	Various
M1	30	M1	Various
M2	30	M2	Various
SR	20	M1, M2	SR
SR+HIP	20	M1, M2	SR+HIP
SR+HIP+HT	20	M1, M2	SR+HIP+HT

The analyses of the data groupings are summarized in the following sections. Single variable analyzes characterize the material properties for a single data grouping. The two-sample comparisons analyze data originating from different groupings, making visual comparisons and testing hypotheses to determine if differences in properties between the groups are statistically significant. The ANOVA analysis compares material properties for multiple groups.

Single Group Analyses

Raw data from the mechanical test reports were used to calculate the mean values of ultimate tensile strength σ_{UTS}, yield strength σ_Y, elastic modulus E, fracture elongation e, and reduction in area A_R for each data grouping. Table 3 summarizes the mean material property values for the groupings.

Table 3 Mean material property data for specimen groups

Grouping	Sample size	Ultimate tensile stress (KSI)	Yield stress (KSI)	Modulus (MSI)	Elongation (%)	Reduction in area (%)
M1+M2	60	184.4	143.1	27.7	25.7	33.9
M1 SR	10	164.7	128.3	22.4	21.2	25.3
M2 SR	10	171.9	129.6	27.4	30.8	42.1
M1 SR+HIP	10	182.8	132.6	28.7	23.0	25.2
M2 SR+HIP	10	186.0	134.3	29.5	28.4	37.3
M1 SR+HIP+HT	10	199.6	166.2	29.1	24.8	35.8
M2 SR+HIP+HT	10	201.7	168.0	29.0	25.8	38.0
M1	30	182.4	142.3	26.7	23.0	28.7
M2	30	186.5	144.0	28.6	28.3	39.1
SR	20	168.3	129.0	24.9	26.0	33.7
SR+HIP	20	184.4	133.5	29.1	25.7	31.2
SR+HIP+HT	20	200.6	167.1	29.1	25.3	36.9

The scatterplots in Figures 1 through 5 graphically illustrate the differences between the group means for tensile strength σ_{UTS}, yield strength σ_Y, elastic modulus E, fracture elongation e, and reduction in area A_R, respectively. From these plots, some trends start to emerge: 1) that identical specimens produced on different machines do seem to have slightly different properties (particularly for σ_{UTS}, σ_Y, and E), 2) that the differences in mean material properties for equivalent specimens built on different machines seem to decrease with subsequent postprocessing (HIP and heat treatment), and 3) that HIP and HT generally improve mechanical properties. Further statistical tests (summarized in the two group comparison and multigroup analyses sections) assess whether the discrepancies in means between groups are statistically significant and if the apparent equalization effect of HIP and heat treatment is real.

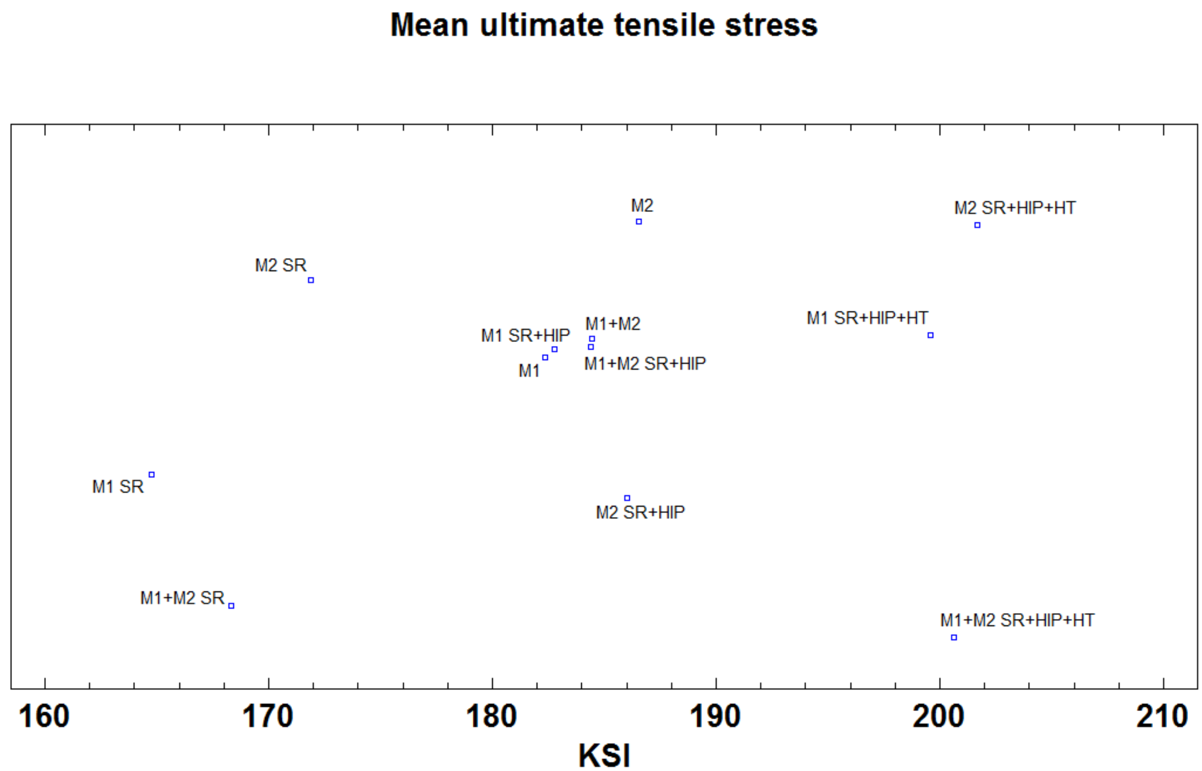

Figure 1 Comparison of mean ultimate tensile stress for data groupings

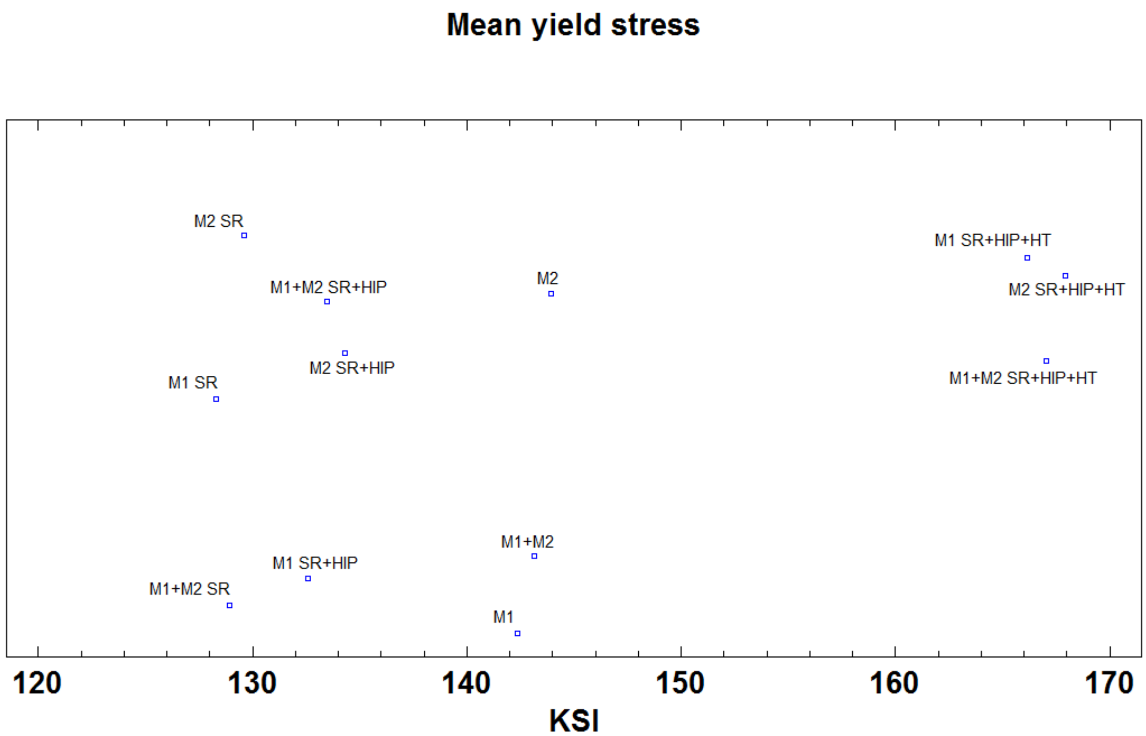

Figure 2 Comparison of mean yield stress for data groupings

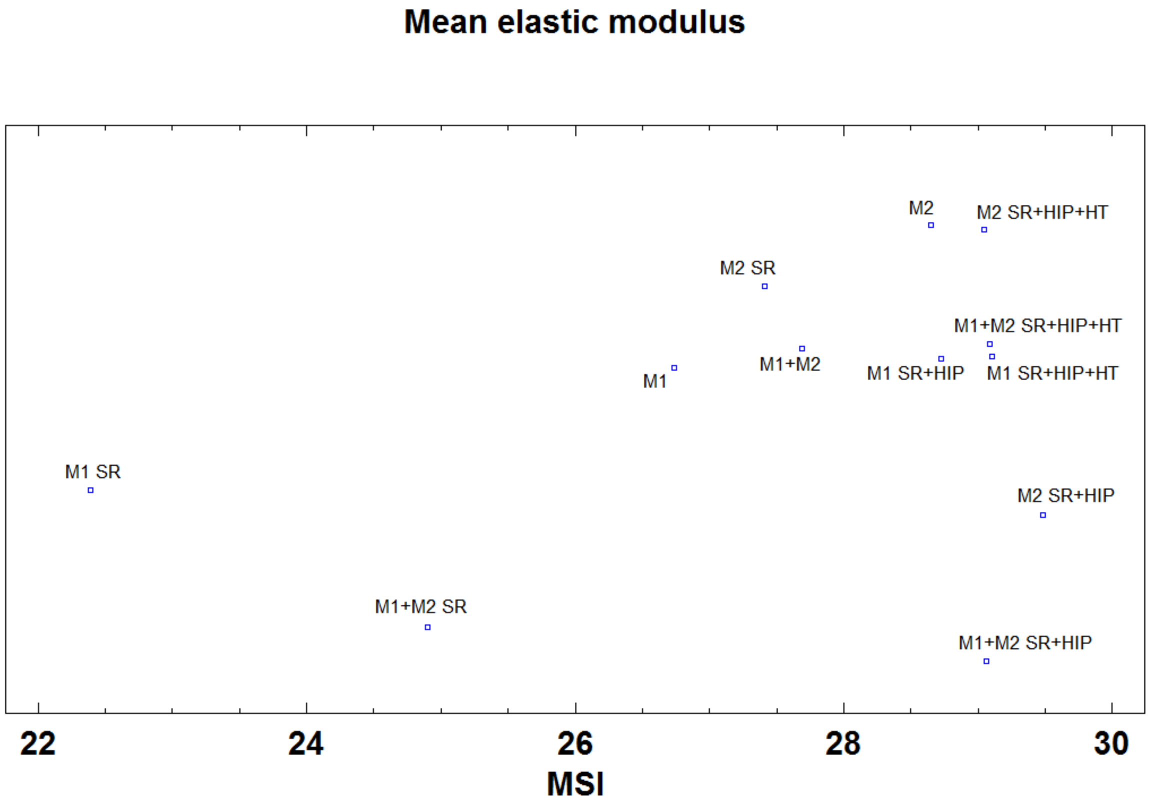

Figure 3 Comparison of mean elastic modulus for data groupings

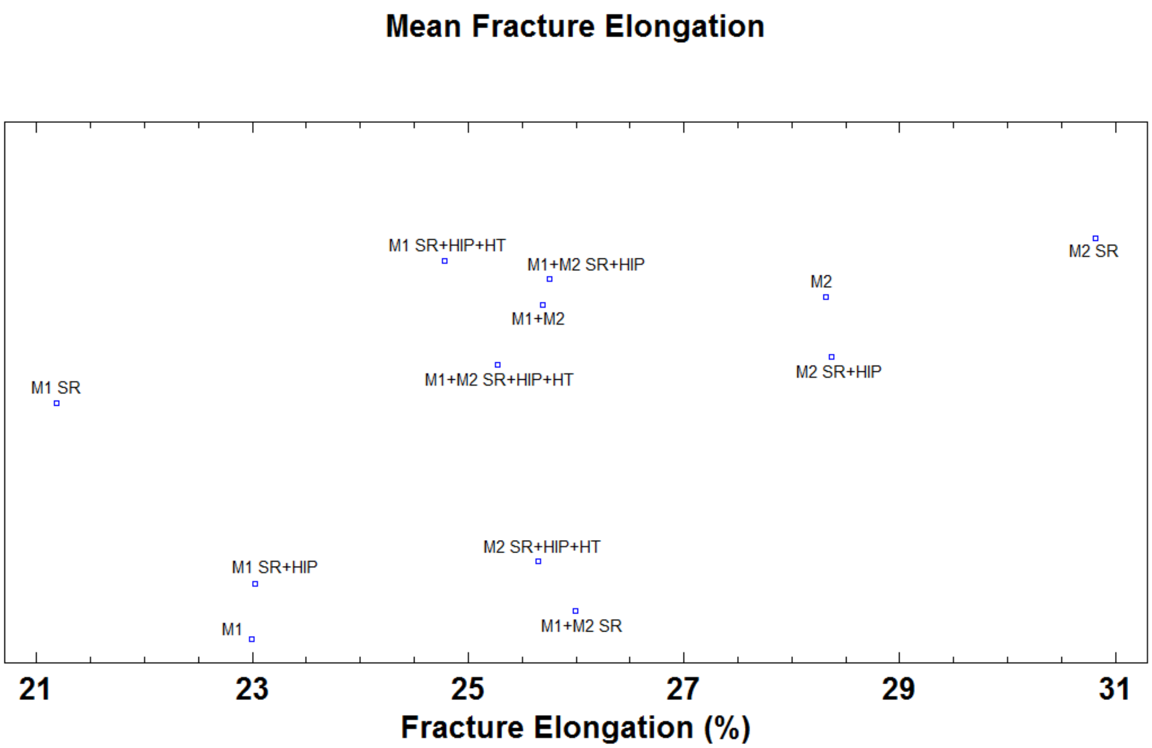

Figure 4 Comparison of mean fracture elongation for data groupings

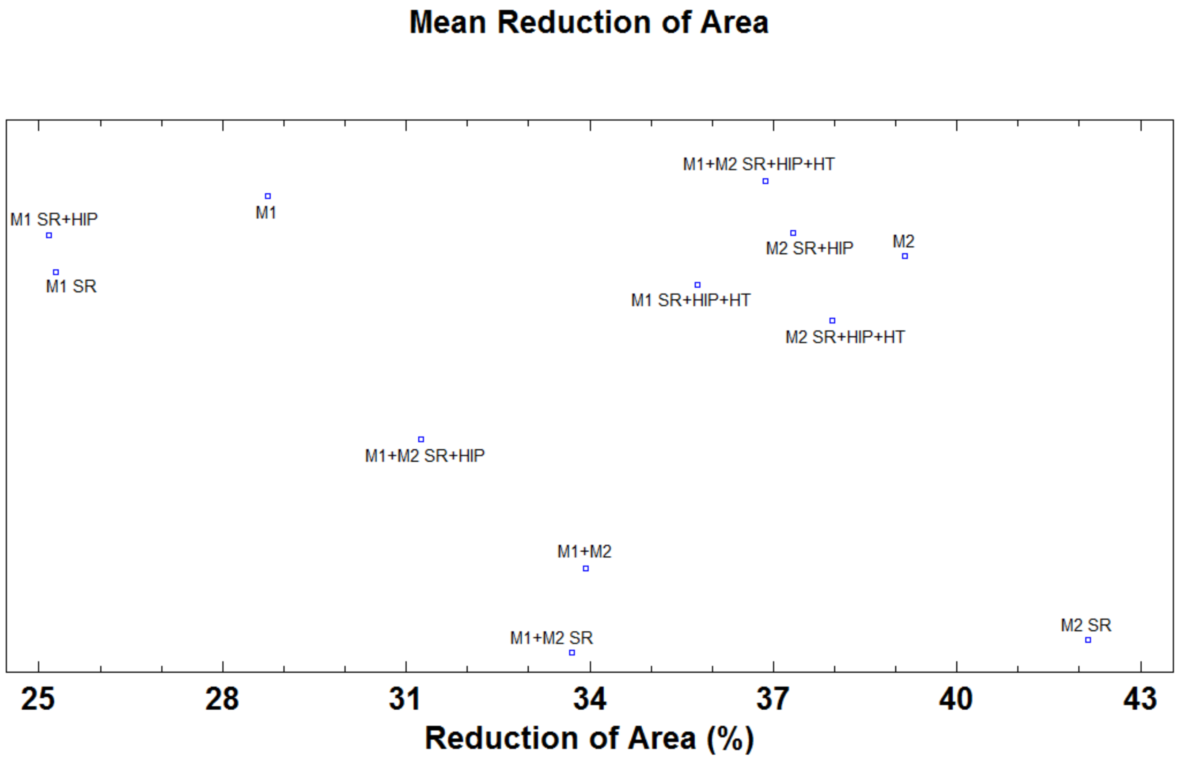

Figure 5 Comparison of mean reduction of area for data groupings

Two Group Comparisons

Two-variable statistical comparative procedures were used to compare the means, medians, and standard deviations for the material properties (ultimate tensile stress σ_u, yield stress σ_y , modulus E, % elongation δ, and % reduction in area A) between selected data. The two-group comparisons and ANOVA (next section) are complementary to the single group analyses. The latter asked the question "what are the differences in material property values for the various data groupings?" The goal of the two variable analyses and ANOVA is to determine whether these differences are statistically significant.

Analyses were restricted to comparisons that would help answer two key questions of interest in this study:

1) Is there a statistically significant difference in material properties for samples which underwent the same heat treatment but were produced on different machines?
2) Is there a statistically significant difference in material properties for samples which were produced on the same machine but underwent different heat treatments/postprocessing regimes?

With these points in mind, the following data groups were compared: M1 SR vs. M2 SR, M1 SR+HIP vs. M2 SR+HIP, M1 SR+HIP+HT vs. M2 SR+HIP+HT, M1 SR vs. M1 SR+HIP, M1 SR vs. M1 SR+HIP+HT, M1 SR+HIP vs. M1 SR+HIP+HT, M2 SR vs. M2 SR+HIP, M2 SR vs. M2 SR+HIP+HT, and M2 SR+HIP vs. M2 SR+HIP+HT.

Analyses were additionally limited to comparison of groups with equal sample sizes. While a large difference in sample size does not invalidate the statistical tests used to make a comparison (normality is the relatively more important underlying assumption), the disparity does create skewness in the t-statistic used to determine whether the difference in means for the groups is statistically significant. We must be particularly cautious of the case where the larger variance is associated with the smaller sample size, since the t-statistic will then be dominated by the variance for the smaller sample [10].

The most valuable comparisons are those which satisfy the following criteria:

a) sample sizes are equal

b) only one variable is different for the data sets (ex. they were produced on the same machine but one set underwent an additional heat treatment or the specimens were subject to the same postprocessing conditions but produced on different machines)

c) the assumption for normality are not violated (kurtosis and skewness metrics are between -2 and 2)

Summary of statistical tests

- Comparison of means

The first formal comparison between the two data sets is to test the hypothesis that the means of the populations from which the data arise are equal (the null hypothesis) versus the alternative hypothesis that they are different. The procedure used to compare the means is the t-test (performed here at the 95% confidence level). If the p-value associated with the test is less than 0.05, there is sufficient evidence to reject the null hypothesis that the means are equal and declare the population means to be statistically different.

- Comparison of variances

We are not only interested in comparing the mean material properties for the specimens, but also want to catalog shifts and differences in variability. The F-test for equivalence of variances is used to detect such differences and determine their statistical significance. The F-statistic is used to test the hypothesis that the variance of the populations from which the two data sets arise are equal. If the p-value associated with the test is less than 0.05, there is sufficient evidence to reject the null hypothesis that the variances are equal and declare the population variances to be statistically different.

- Comparison of medians

While the mean is the statistical metric most frequently used in characterization and summary of data, its primary drawback is its sensitivity to outliers in the data set which have a tendency to skew the average to a higher or lower value (and, depending on the number and degree of the outliers, may result in a value that is not truly representative of the data). The median, in contrast, is an unbiased estimator that will not be affected by the presence of outliers (an outlier is defined as a point that lies beyond 3 standard deviations of the mean).

The Mann-Whitney test for equivalence of means is similar to the t-test. A p-value less than 0.05 leads us to conclude that the medians of the two populations from which the samples originate are significantly different [11].

- Comparison of distributions

The Kolmogorov-Smirnoff test compares the maximum vertical distance between the cumulative probability distribution for two samples to assess whether they are likely to have originated from significantly different populations [11]. As a nonparametric test, the K-S test does not require an assumption of normality and can thus be applied to the pooled data sets in

the variability study (which have non-normal characteristics) without reservation. The K-S test is also less sensitive to small sample sizes and/or disparities in sample size between data sets.

The p-value associated with the K-S statistic is used to determine whether the population distributions associated with the samples being compared are significantly different (yet makes no assumptions about the shape, symmetry, or parameters of the distributions). A p-value of less than 0.05 leads to the conclusion that there is a significant difference at the 95% confidence level. Conversely, a p-value of greater than 0.05 means that there is not enough evidence to reject the null hypothesis that the samples originated from the same population distribution. A visual tool for interpreting the results of the K-S test is the quantile plot.

Discussion and conclusions (two group comparisons)

Key observations from the two variable analyses that appear consistently are:

1) Identical specimens built at the same parameters and subject to the some postprocessing and heat treatment protocols but produced on different machines can have material properties that are different at a statistically significant level. This is demonstrated by the group comparisons M1 SR/M2 SR and M1 SR+HIP/M2 SR+HIP, for which the mean, median, standard deviation, and population distribution are typically different for each of the five properties evaluated (although the HIP step seems to erode the differences in modulus). This is not the case for the M1 SR+HIP+HT/M2 SR+HIP+HT comparison (see point 3). Table 4 tabulates the properties which were determined to be different (at the 0.05 level) for each statistical metric in the M1 SR/M2 SR and M1 SR+HIP/M2 SR+HIP comparisons. Table 5 indicates the average percent difference in each property for the two groups.

Table 4 Statistically Significant Differences ($p<.05$) Detected in Group Comparisons

Comparison	Means	Variance	Median	Distribution
M1 SR vs M2 SR	σ_u, σ_y, E, e, A_r	σ_u, e, A_r	σ_u, σ_y, E, e, A_r	σ_u, σ_y, E, e, A_r
M1 SR+HIP vs M2 SR+HIP	σ_u, σ_y, e, A_r	σ_u, e, A_r	σ_u, σ_y, e, A_r	σ_u, σ_y, e, A_r

Table 5 Summary of Percent Difference in Mean Material Properties for Group Comparisons

Comparison	σ_u	σ_y	E	e	A_r
M1 SR vs M2 SR	*4.3*	*1.0*	*22.0*	*45.4*	*66.8*
M1 SR+HIP vs M2 SR+HIP	*1.8*	*1.3*	*2.6*	*23.2*	*48.4*

2) For groups of identical specimens produced on the same machine but where one group is subject to additional postprocessing (HIP) or heat treatment, the general effect of these processes is a statistically significant improvement in material properties. This conclusion is illustrated by the following comparisons: M1 SR vs M1 SR+HIP, M1 SR+HIP vs M1 SR+HIP+HT, M2 SR vs M2 SR+HIP, M2 SR+HIP vs M2 SR+HIP+HT. Table 6 tabulates the properties which were determined to be different (at the 0.05 level) for each statistical metric in these comparisons.

Table 6 Statistically Significant Differences ($p<.05$) Detected in Group Comparisons

Comparison	Means	Variance	Median	Distribution
M1 SR vs M1 SR+HIP	σ_u, σ_y, E	σ_u, A_r	σ_u, σ_y, E	σ_u, σ_y, E
M1 SR+HIP vs M1 SR+HIP+HT	σ_u, σ_y, A_r	σ_y, e	σ_u, σ_y, A_r	σ_u, σ_y, A_r
M1 SR vs M1 SR+HIP+HT	σ_u, σ_y, E, A_r	σ_y, E, e	σ_u, σ_y, E, A_r	σ_u, σ_y, E, A_r
M2 SR vs M2 SR+HIP	σ_u, σ_y, E, e, A_r	E	σ_u, σ_y, E, e, A_r	σ_u, σ_y, E, e, A_r
M2 SR+HIP vs M2 SR+HIP+HT	σ_u, σ_y, e	σ_u, , σ_y, e, A_r	σ_u, σ_y, e	σ_u, σ_y, e
M2 SR vs M2 SR+HIP+HT	σ_u, σ_y, E, e, A_r	σ_u, σ_y	σ_u, σ_y, E, e, A_r	σ_u, σ_y, E, e, A_r

What degree of property improvement do the HIP and heat treatment steps impart? Table 7 summarizes the difference in each of the mean material properties for the comparison (Table 6 indicates whether these changes are statistically significant).

Table 7 Summary of Percent Difference in Mean Material Properties for Group Comparisons

Comparison	σ_u	σ_y	E	e	A_r
M1 SR vs M1 SR+HIP	*11.0*	*3.3*	*28.3*	*8.7*	*-0.4*
M1 SR+HIP vs M1 SR+HIP+HT	*9.2*	*25.3*	*1.25*	*7.6*	*42.2*
M1 SR vs M1 SR+HIP+HT	*21.1*	*29.5*	*29.9*	*17.0*	*41.5*
M2 SR vs M2 SR+HIP	*8.25*	*3.7*	*7.6*	*-7.9*	*-11.4*
M2 SR+HIP vs M2 SR+HIP+HT	*8.3*	*25.0*	*-1.5*	*-9.2*	*1.7*
M2 SR vs M2 SR+HIP+HT	*17.3*	*29.6*	*5.6*	*-16.4*	*-9.9*

If we look at successive plots of the data distributions as the samples go through HIP and heat treatment, we can clearly see the observed trend of strength property improvement with additional processing. As an example, Figures 6 and 7 display a scatterplot and a box and whisker plot, respectively, of ultimate tensile stress data for the groups M1 SR, M1 SR+HIP, and M1 SR+HIP+HT. This property improvement also extends to yield strength and elastic modulus. A similar trend is observed for the analogous M2 data.

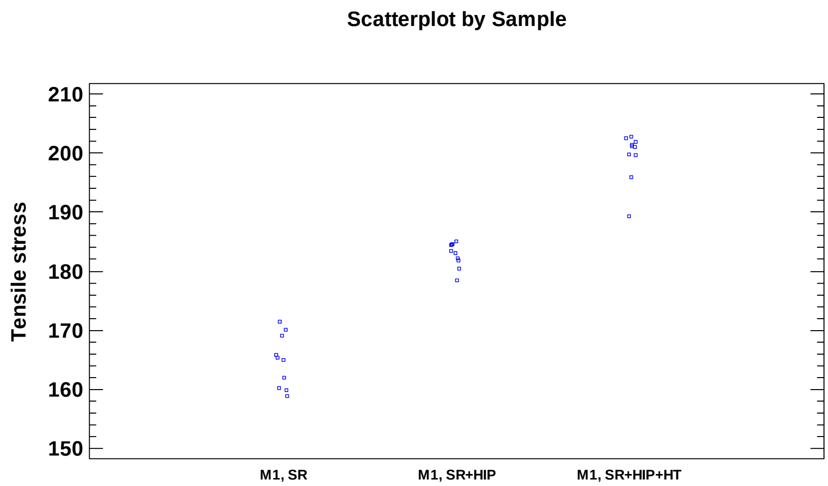

Figure 6 Scatterplot comparing ultimate tensile stress data (in KSI) for M1 SR, M1 SR+HIP, and M1 SR+HIP+HT.

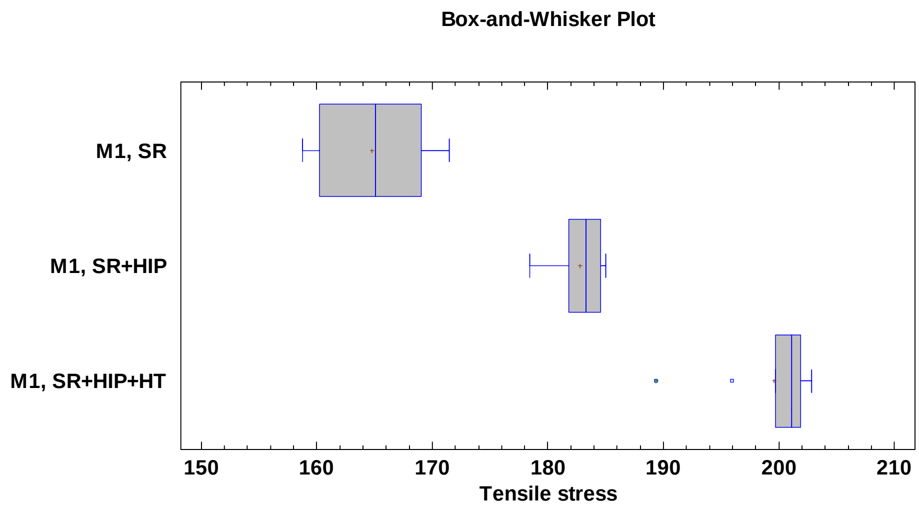

Figure 7 Box and whisker plot comparing ultimate tensile stress data (KSI) for M1 SR, M1 SR+HIP, and M1 SR+HIP+HT.

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Metallurgical characterization of samples built on the same machine but at different stages in the process (i.e. as-built, HIP, and HIP + subsequent heat treatment) is essential to shed light on the microstructural evolution which imparts the observed statistically significant changes in properties. HIP can reduce or eliminate the presence of pores – this improved material consolidation may contribute to enhanced mechanical performance, as HIP is associated with a large increase in yield strength for the machine variability data. Material changes which take place during HT harden the material and seem to increase the ultimate tensile strength. This hardening may have an ancillary effect of making the specimen more brittle, reducing the percent elongation values (although this is not the case for the M2 data). The broad conclusion from this comparison is that HIP does more to improve yield strength, while HT has a more significant impact on ultimate tensile strength.

There are some anomalous results which could potentially be explained by metallurgical evaluation:

a) The large increase in modulus with additional processing which was observed for the M1 specimens but not for M2
b) Why percent elongation and area reduction increase as the M1 specimens go through HIP and HT, but decrease for the M2 specimens
c) For identical specimens built at the same parameters but on different machines, postprocessing can narrow the initial property differences observed in the as-built samples to a level that is no longer statistically significant. This is apparent in Table 8, which indicates the differences in properties which are significant at the .05 level for the M1 SR/M2 SR, M1 SR+HIP/M2 SR+HIP, and M1 SR+HIP+HT/M2 SR+HIP+HT comparisons. While the as-built (SR) specimens are characterized by significant differences across properties (σ_u, σ_y, E, e, A_r) and statistical metrics (mean, variance, median, distribution), the specimens which have gone through HIP and heat treatment do not exhibit these differences.

Table 8 Statistically Significant Differences ($p<.05$) Detected in Group Comparisons

Comparison	Means	Variance	Median	Distribution
M1 SR vs M2 SR	σ_u, σ_y, E, e, A_r	σ_u, e, A_r	σ_u, σ_y, E, e, A_r	σ_u, σ_y, E, e, A_r
M1 SR+HIP vs M2 SR+HIP	σ_u, σ_y, e, A_r	σ_u, e, A_r	σ_u, σ_y, e, A_r	σ_u, σ_y, e, A_r
M1 SR+HIP+HT vs M2 SR+HIP+HT	----	A_r	----	----

Table 9 catalogs the percent difference in the mean material properties for the data groups being compared. It is apparent that the difference in mean material properties is reduced by the HIP process and further reduced by heat treatment. While the ultimate tensile strengths and yield strengths for the M1 SR and M2 SR specimens initially have the smallest discrepancy, these differences were found to be significant at the 0.05 level (see the corresponding entry in Table 8). The large disparity in modulus, elongation, and area reduction "shrinks" with HIP; following heat treatment, mean differences in mechanical material properties for the M1 and M2 specimens were not found to be significant.

Table 9 Summary of Percent Difference in Mean Material Properties for Group Comparisons

Comparison	σ_u	σ_y	E	e	A_r
M1 SR vs M2 SR	*4.3*	*1.0*	*22.0*	*45.4*	*66.8*
M1 SR+HIP vs M2 SR+HIP	*1.8*	*1.3*	*2.6*	*23.2*	*48.4*
M1 SR+HIP+HT vs M2 SR+HIP+HT	*1.0*	*1.1*	*-0.13**	*3.9*	*6.1*

*negative value means that M2 group mean is greater

The equalization effect of postprocessing and heat treatment in reducing machine variability can be visualized using box and whisker plots. As an example, we examine the evolution of yield strength for specimens built on the M1 and M2 machines as they undergo HIP and heat treatment. Figure 8 shows the box and whisker plot for the yield strengths of M1 SR, M2 SR, M1 SR+HIP, M2 SR+HIP, M1 SR+HIP+HT, and M2 SR+HIP+HT. Mean yield strength improves for both M1 and M2 specimens with additional processing and the amount of overlap between the M1 and M2 data sets decreases to a point that the differences between the data sets are no longer statistically significant.

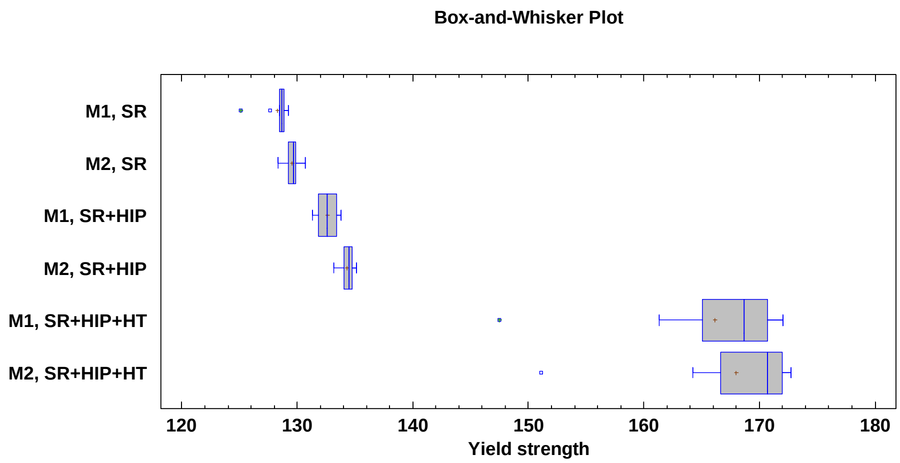

Figure 8 Comparison of means for yield strength (KSI)

It is clear from the two-group analyses that HIP and heat treatment are necessary not only to improve properties (specifically to elevate the properties of additively manufactured materials to values that resemble their cast or wrought counterparts), but also to reduce the effects of process variability. This observation is confirmed by the ANOVA summarized in the next section.

Multigroup Analyses

Comparison of multiple groups were performed using the ANOVA (analysis of variance) technique to test the (null) hypothesis of equal population means. Thus rejection of the null hypothesis (which occurs when $p<0.05$) in this context indicates that the samples originate from populations whose means are significantly different.

While the results of the ANOVA are (and ideally should be) a confirmation of the two group analyses, it does not render the latter unnecessary. The danger in making comparisons across many pairs of means is that the probability of an error on at least one pair increases, which is why it is important to also perform the two variable comparisons previously summarized in addition to the ANOVA.

Material properties for the following data groupings were compared using ANOVA: M1 SR, M2 SR, M1 SR+HIP, M2 SR+HIP, M1 SR+HIP+HT, and M2 SR+HIP+HT. The analysis considers the combined effect of machine variability and differences in post-processing/heat treatment. This comparison represents the most succinct and compelling summary of the variability data, as they show simultaneously how properties vary with equipment and postprocessing conditions.

Examining the data among the six groups simultaneously, we note several key trends:

- The effect of HIP and heat treatment at reducing the effect of machine variability on material properties is clearly illustrated by the means and quantile plots. The means plots for ultimate tensile strength, yield strength, and modulus show that the amount of overlap between the M1 and M2 sample groups increases with HIP and that the intervals which bound the means are nearly coincident for the M1 SR+HIP+HT and M2 SR+HIP+HT data sets. This equalization effect was noted previously in the two variable comparative analyses, but is confirmed further by the ANOVA.

  As an example of this, the means plots for ultimate tensile strength for the multi-group comparison is displayed in Figure 9. The bars which bound the mean (denoted by a

circle) represent the uncertainty intervals. The amount of overlap between the group means for the M1 and M2 groups increases with additional processing.

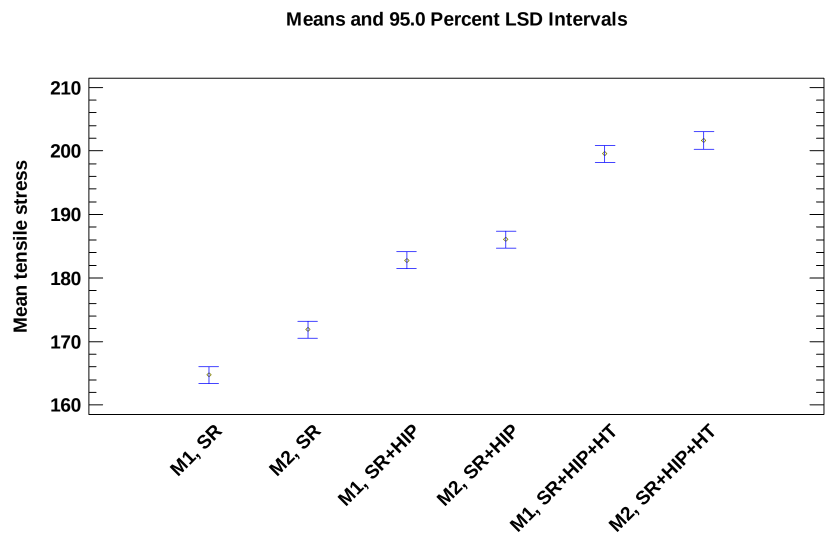

Figure 9 Means plot for ultimate tensile stress (KSI)

- The distance between the cumulative probability distributions for the data sets M1 and M2 "shrink" with HIP and decrease further with heat treatment, which can be seen on a quantile plot. This graph plots the proportion of data in each sample that is below a given value of X, as a function of X. The function is cumulative (ex. the x value corresponding to a value of 1 is the upper limit of the data since 100% of the samples fall below it). Quantile plots which lie very close together indicate that the samples come from the same population/distribution, while an offset of one plot relative to another indicates that the distributions are different. The Kolomogorov-Smirnoff test, which characterizes the statistical significance of the maximum distance between quantiles of the data groups being compared, is visually represented by these plots. The quantile plot for mean ultimate tensile strength is shown in Figure 10. Statistically, this narrowing of the gap between lines on the quantile plot with additional processing (HIP+HT) means it is more likely that the data sets being compared originate from the same population distribution (a visual observation confirmed by the results of the Kolmogorov-Smirnoff test).

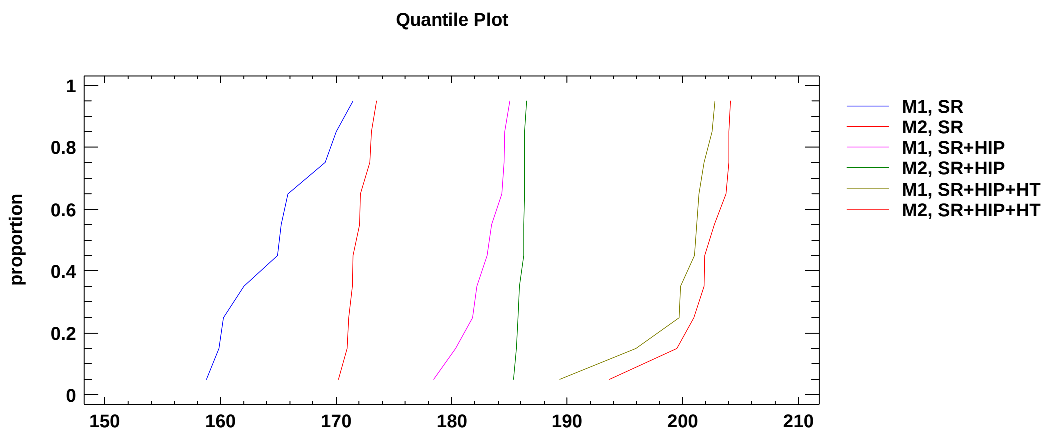

Figure 10 Quantile plot for mean ultimate tensile stress (KSI)

- Trends in changes in percent elongation and area reduction with heat treatment and machine are less apparent (although the differences in means among the 6 groups are still significant at $p=0.05$). There is substantial vertical overlap for these properties among data groups in the means plot (Figure 11 shows the percent elongation plot as an example). The quantile plot is characterized by crossover (which indicates a failure to accept the K-S alternative hypothesis that the population distributions for the sample sets are significantly different). Figure 12 shows the quantile plot for percent area reduction among the six groups.

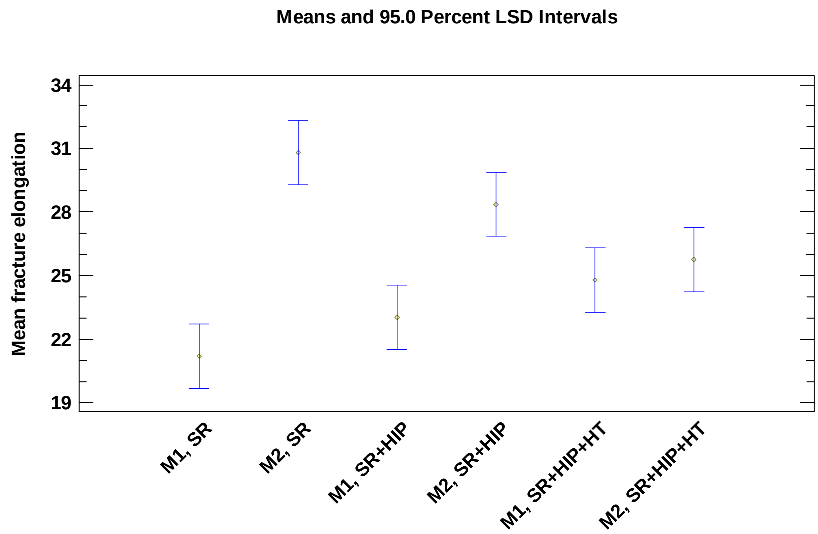

Figure 11 Means plot of percent fracture elongation

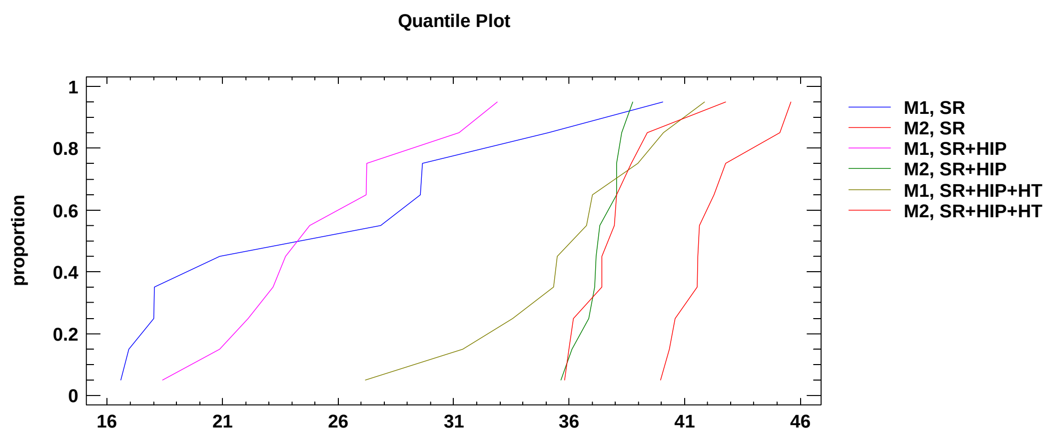

Figure 12 Quantile plot of percent area reduction

Conclusions and Future Work

The key conclusions of the study presented here are:

1) It is possible of identical samples which underwent the same heat treatment but were produced on different machines to exhibit a statistically significant difference in characteristic mechanical material properties.
2) In general, there is a statistically significant difference in characteristic mechanical material properties for samples which were produced on the same machine but underwent different heat treatments/postprocessing regimes. This difference usually represents a property improvement.
3) While the purpose of HIP and heat treatment is to improve mechanical properties by reducing porosity and increasing strength, the postprocessing regime also has the ancillary benefit of eroding the impact of machine variability on properties to a level that, in the case of the specimens which underwent both HIP and heat treatment, its impact on group means is no longer statistically significant.

As noted in the two group analyses discussion, a follow-on investigation of the specimens in the machine variability study should include optical microscopy of sample cross-sections and/or SEM analysis. Microscopic evaluation will provide insight into what specific changes in the microstructure brought about by additional processing are responsible for the a) the equalization effect (where variability in mechanical properties for the M1 and M2 groups is reduced by HIP and further reduced by heat treatment). Microscopy can also potentially explain some of the anomalous/nonintuitive trends in the data (such as why a large increase in modulus with additional processing was observed for the M1 specimens but not for M2 or why percent elongation and area reduction increase as the M1 specimens go through HIP and HT, but decrease for the M2 specimens).

A second machine variability study is currently planned to characterize how machine variability impacts mechanical performance in environments that are of interest to the propulsion community. This study will include an addition 60 samples that will be tested in liquid nitrogen, liquid hydrogen, and at both elevated and cryogenic temperatures characteristic of operational propulsion systems.

A round robin study for machine variability should also be a priority for further research activities. As AM techniques are increasingly adopted by government and industry to produce propulsion hardware in human-rated systems, development work is needed to establish these materials as reliable alternatives to conventional subtractive manufacturing. Machine variability,

based on this and other studies, has been recognized as a factor that influences properties of additively manufactured parts and that baseline material properties might not be maintained when parts are produced on different machines. For future programs, NASA will procure parts from vendors who use a diverse range of machines to produce parts and it is essential that NASA (as well as the broader AM community) develop a sound understanding of degree to which machine variability influences material properties. A round robin study would characterize variability between machines used by vendors and NASA MSFC AM laboratory. In this class of study, vendors, original equipment manufacturers (OEMs) of AM machines, and NASA would produce parts of the same material and geometry at the same parameters, but on a wide range of machines. Such work is also needed to assess whether machine variability effects are more pronounced in parts built using powder bed fusion (PBF) techniques, where implementation of robust process controls is perhaps more difficult than for wire fed systems.

In summary, the aerospace community faces unique challenges that must be addressed before the potential design and economic benefits of AM can be realized. Currently AM parts are process specific and standards are required to assess sensitivity of parts to base material (powder) composition, address machine variability, establish post-processing controls (i.e. heat treatment and surface treatment protocols), and ultimately ensure that AM products are safe and reliable prior to their deployment in crewed systems. As we have demonstrated, even with robust process controls, it is possible for two AM machines operating at identical parameters with the same base material to produce parts with slightly different material properties. While understanding machine variability is only one link of many in the chain to establishing "certify as you build" process robustness, its characterization is critical to design, analysis, hardware acceptance and certification of AM parts. Though AM technologies will not supplant manufacturing for high rate production, low cost parts anytime soon, it is attractive for low rate complex geometries that are characteristic of rocket propulsion hardware. To leverage AM's promise for creating affordable propulsion systems, we must undertake the foundational work necessary to understand the process and mitigate risk associated with replacing conventional subtractive manufacturing techniques with AM in human rated aerospace systems.

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